



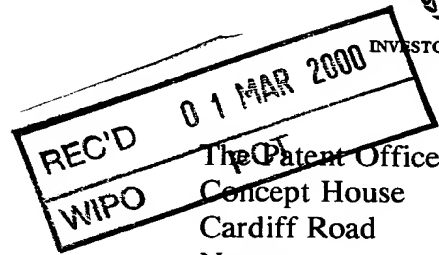
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Signed

Andrew Gersey

Dated

18th February 2000



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JAN99 1417522-1 000107  
P01/7700 0.00 - 9900654.6

# Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

THE PATENT OFFICE

14 JAN 1999

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The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

1. Your reference

M99/0009/GB

2. Patent application number

(The P

9900654.6

14 JAN 1999

3. Full name and postcode of the or of each applicant (underline all surnames)

Reflective Technology Industries  
Limited  
Road One  
Winsford Industrial Estate  
Winsford  
Cheshire CW7 3QQ

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

GB

6449227003

4. Title of the invention

Retroreflective Inks

5. Name of your agent (if you have one)

McNeight & Lawrence

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Regent House  
Heaton Lane  
Stockport  
Cheshire SK4 1BS

Patents ADP number (if you know it)

0001115001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

Yes

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an applicant, or
  - c) any named applicant is a corporate body.
- See note (d))

**Patents Form 1/77**

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description 18

Claim(s) 7

Abstract

Drawing(s) 1

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11.

I/We request the grant of a patent on the basis of this application

Signature

Date 13.1.99

McNeight & Lawrence

12. Name and daytime telephone number of person to contact in the United Kingdom

David L McNeight 0161 480 6394

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## RETROREFLECTIVE INKS

This invention relates to retroreflective inks and methods for making them.

Retroreflective coating compositions have been the subject of numerous patents, for example US Patents 2 963 378, Palmquist *et al*, 3 099 637, 3 228 897 and 3 420 597, Nellessen, 3 535 019, Longlet *et al* and 4 103 060 and 4 263 345, Bingham *et al*. A retroreflective ink has been commercially available for a number of years, marked by the 3M company, this product being available in dark grey and sold as a three pack system, comprising a binder dispersion system, a pack of hemispherically coated glass microspheres or beads and a coupling agent, which are mixed just prior to use.

One pack inks were proposed in WO 94/06869, M N Ellis and in EP 0 729 592, Reflective Technology Industries Limited and US 5 650 213, Reflective Technology Inc., which also disclose the incorporation of pigment. US 5 650 213 specified a range of pigment particle size which is what is, in fact, the usual range commercially available, and ranges of binder/bead and binder/(bead and pigment) volume ratios which are seemingly the ranges of choice to produce an ink which is printable by conventional screen printing techniques.

Problems associated with the performance of reflective coatings, especially in the convenient, one-pack form that does not require mixing just prior to printing, involve shelf life, washfastness and abrasion resistance. These key areas are interrelated - the binder system must be such as will not allow the beads to settle even over extended storage periods, and it must also not couple to the beads during that storage, yet it must, on printing, adhere the beads to the substrate in a reasonably washfast and abrasion resistant manner while permitting the beads to be exposed appropriately to retroreflect light.

The severity of these problems may explain the sale by 3M of the three pack system and the fact that the inks produced according to US 5 650 213 are formulated solely for the production of printed fabric by the patentee Reflective Technology Inc. and not for sale to printers.

The present invention addresses these - and other - problems and provides long shelf life one pack retroreflective ink systems with good washfastness and abrasion resistance.

The invention, in one aspect, comprises a one-pack retroreflective ink comprising microbeads in a liquid carrier medium including binder chemicals for attaching the microbeads to a substrate to which the ink is to be applied, and a coupling agent which couples the microbeads and cross-links the binder chemicals, characterised in that the coupling agent is unreactive except at elevated temperature at which the ink on the substrate is cured.

The invention, in another aspect, comprises a one-pack or a two-pack retroreflective ink comprising microbeads in a liquid carrier medium including binder chemicals for attaching the microbeads to a substrate to which the ink is to be applied, the microbeads being incorporated in the carrier medium, and a coupling agent which couples the microbeads and cross-links the binder chemicals, characterised in that the coupling agent is not activated until the ink is printed.

The two-pack system comprises a separate pack for the coupling agent.

The ink may comprise retroreflective and/or non-retroreflective microbeads.

The binder system may comprise polyvinylidene chloride copolymer and the coupling agent comprise (3-aminopropyl) silanetriol.

The binder system may comprise polyvinylidene chloride copolymer and the coupling agent comprise blocked 1, 6 hexamethylene diisocyanate trimer.

The binder system may comprise polyurethane and the coupling agent comprise blocked 1, 6 hexamethylene diisocyanate trimer.

The microbeads may have an aluminium coating, and may be pre-treated with a silicate before inclusion in the ink. They may be pretreated with sodium silicate. They may be treated with an amino silane, which treatment may be after a silicate treatment and before inclusion in the ink. The amino silane may be bis-[*gamma*-(trimethoxysilyl) propyl] amine.

The ink may comprise pigment, and may, especially when comprising pigment, comprise non-retroreflective, which usually means un-metallised, beads.

The microbeads may be pretreated before metallisation with stannous chloride.

The ink may be formulated - as to, e.g. their viscosity, particle size - suitably for screen printing. The microbeads may be 40 micron beads, and are, for best retro-reflectivity, of high, e.g. 1.9, refractive index glass.

The ink may comprise a humectant, which may comprise urea and/or 2,3 propanediol, and may be water-based. It may comprise a buffer, to ensure an appropriate pH, such buffer, for example, comprising an ammonium phosphate buffer or a sodium

phosphate buffer. A dispersant may also be included, as may a defoamer, a thickening agent, a cross-linking agent and a softening agent.

Non-water based inks may also be comprised within the invention. In this case, the need to protect the aluminium coating against attack in water-based media may be less important.

Surprisingly, having regard to the teaching of US 5 650 213, substantially better quality inks - in terms of reflectivity, washfastness, abrasion resistance and shelf life - are produced with binder to bead volume ratios equal to or less than 50%. Essentially, more beads can be attached using less obscuring binder, more firmly and more permanently than when the prior art binder to bead ratios are used.

For a screen printing ink, the viscosity is desirably equal to or less than 40 pascals at room temperature.

The invention also comprises a method for making a one-pack retroreflective ink comprising the steps of:

- making microbeads;
- suspending the microbeads in a liquid carrier medium;
- the liquid carrier medium comprising binder chemicals for attaching the microbeads to a substrate to which the ink is to be applied and a coupling agent which couples the microbeads and cross-links the binder chemicals, the coupling agent being unreactive except at elevated temperature at which the printed substrate is cured.